

Please amend the application as follows:

**In the Claims:**

Please cancel Claim 6. Please amend Claim 1, and add new Claims 75-77 as indicated in the attached Claims listing.

**REMARKS**

In accordance with 37 C.F.R. § 1.121, a complete listing of all claims in the application is attached.

Claims 1- 5 and 7-10 remain in the application. Claim 6 has been canceled. Claims 75-77 have been added. The following remarks are offered in support of allowance of Claims 1- 5, 7-10, and 75-77. Reexamination and reconsideration of the application in light the following remarks are respectfully requested.

Claim 1 has been amended by deleting the reference to nootkatone. Support for this amendment is found, for example, in Claims 1-5 and 7-10, as originally filed.

Independent claim 75 has been added. Claim 75 is directed to treatment of a material with an effective amount of nootkatone such that the treated material repel ants. Support for this independent claim is found, for example, in the Specification, §§ [0002], [0021], [0022], [0027] and [0030]-[0041], and in Claims 1, 3, and 6 as originally filed.

Claims 76 and 77 depend from Claim 75. Support for these new dependent claims is found, for example, in the Specification, §§ [0002], [0021], [0022], [0027] and [0030]-[0041], and in Claims 1, 3, 5, and 6 as originally filed.

***The §102 Rejections***

***Zobitne et al.*** Claims 1, 4-6, and 10 were rejected under 35 U.S.C. §102(a) as being anticipated by *Zobitne et al.*, U.S. Patent No. 6,548,085. As stated by the Office, *Zobitne* teaches a method of applying grapefruit oil to the pavement to kill ants." Applicants respectfully disagree that *Zobitne* teaches a method of applying grapefruit oil to the pavement to kill ants. *Zobitne* teaches spraying an aqueous solution of grapefruit oil directly on certain insects, including pavement ants. See, Fig. 24, Col. 4, lines 50-52; Col. 6, lines 25-31; and Col. 18, lines 44 to 64, Table 24.

Claim 1, as amended, is no longer directed to nootkatone. Thus Applicants respectfully submit that this rejection should be withdrawn.

Moreover, Applicants submit that new Claims 75-77 are not anticipated by the Zobitne patent. The Zobitne patent discloses use of these natural oils as insecticides, not repellants. See, for example, Col. 2, line 39, to Col. 3, line 40. This insecticide works by spraying the oil in aqueous solution, preferably with sodium lauryl sulfate, directly on the insect, including directly on pavement ants. See for example, col. 5, lines 16-19; Col. 6, lines 12-31; and Claims 1, 25, and 51. These natural oils are stated to be more effective when sodium lauryl sulfate is added. See, for example, Col. 5, lines 25 to 48; and Col. 18, line 65, to Col. 19, line 4.

Claim 75 is directed to the use of nootkatone to add to material to repel ants. The Zobitne patent does not teach or suggest that grapefruit oil or nootkatone could be added to a material to repel ants. There is no suggestion that grapefruit oil repels ants. Many insecticides kill without repelling the insect; in fact, some are designed to specifically attract insects. Applicants respectfully submit that Claim 75 is not anticipated by the Zobitne patent.

Furthermore, Applicants respectfully submit that the Zobitne patent does not teach or suggest the invention as described in Claim 1, as amended, or Claim 75. Claim 1 is directed to a method to treat material with "an effective amount of a compound selected from the group consisting of  $\alpha$ -cedrene, zizanol, and bicyclovetivenol" to repel or kill ants. (Claim 1). Claim 75 is directed to a method to treat material with nootkatone to repel ants. Applicants respectfully submit that a reference that teaches spraying grapefruit oil directly on ants as an insecticide neither teaches or suggests either that the named compounds in Claim 1 would be effective in either repelling or killing ants, or that nootkatone as in Claim 75 would be effective in repelling ants.

The Office indicates that grapefruit oil is known to contain nootkatone. However, grapefruit oil is a mixture of many compounds. To support this statement, applicants submit to the Office the attached reference that indicates the composition of grapefruit oil. (See D. Sun and P.D. Petracek, "Grapefruit gland oil composition is affected by wax application, storage temperature, and storage time," J. Agric. Food Chem., vol. 47, pp. 2067-2069.) Table 2 of this reference lists about 25 compounds, and indicates that the most abundant

compound in grapefruit oil is limonene, 89 to 93%. Limonene is a known insecticide. See, U.S. Patent Nos. 6,130,253 and 5,977,186, cited in the Specification at ¶ [0016], and submitted with the IDC filed with the application. Nootkatone is only 0.28 to 0.43% of the grapefruit oil. There is no indication in this reference that  $\alpha$ -cedrene is a component of grapefruit oil. Thus, based on the Zobitne patent, a person skilled in the art would neither be motivated to try nor have any expectation that nootkatone or  $\alpha$ -cedrene would repel ants. This patent, which teaches grapefruit oil can kill pavement ants, would neither teach nor suggest that a very small constituent of grapefruit oil (e.g., nootkatone) would repel ants.

*Nakatusu et al.* In addition, the Office rejected Claims 1, 4, 7, and 10 as anticipated by the same reference, citing the Nakatusu *et al.* (U.S. Patent 5,965,518) as pointing "out that grapefruit oil comprises cedrene. See column 4 Table 1, column 3 line 38." Applicants respectfully disagree with this reading of the '518 patent. This patent contains various lists of compounds and essential oils. At column 3, line 38, cedrene is included in a list of "non-aromatic terpenoid compounds." See column 3, lines 37-45. Although the reference indicates that these non-aromatic terpenoid compounds can be components in essential oils, no where does the reference state that cedrene is a component of grapefruit oil. Moreover, the reference later lists essential oils that include "non-aromatic terpenoids as the major constituent." (Column 3, line 66, to Column 4, line 4) Although this listing includes citrus oil, it names only orange oil specifically. Thus this list neither teaches nor suggests that cedrene is a component of grapefruit oil. Column 4, Table 1, lists the components of a certain fragrance that includes Grapefruit oil terpenes. It does not state nor suggest that cedrene is a component of grapefruit oil. In addition, Applicants attach a reference that indicates the composition of grapefruit oil. (See D. Sun and P.D. Petracek, "Grapefruit gland oil composition is affected by wax application, storage temperature, and storage time," J. Agric. Food Chem., vol. 47, pp. 2067-2069.) This reference does not list cedrene as a compound that was found in grapefruit oil.

Thus Applicants respectfully submit that this rejection should be withdrawn.

*Mullen.* Claims 1, 3-7, and 10 were rejected under 35 U.S.C. §102(a) as being anticipated by Mullen, U.S. Patent No. 6,270,753. The Office cited Mullen as teaching "a method of applying grapefruit oil to paper to repel ants." The Office then relies on the

Specification and Nakatsu to indicate that grapefruit oil contains nootkatone and cedrene.

Applicants respectfully submit that Mullen does not anticipate either Claim 1, as amended, or new Claim 75. Mullen does not state that grapefruit oil will kill or repel ants. Instead, it gives a list of natural insect repellants, which includes grapefruit oil along with 11 other compositions. (Col. 3, lines 17-21). It also teaches formulating a fragrance with more than one insect repellant, and then lists insects that "can be repelled" which include ants, along with flies, mosquitoes, and yellow jackets. (Col. 3, lines 14-16). The only example of a formulated reference in Example 1 does not include grapefruit oil. (Col. 4, lines 10 - 40.) No results are reported that would indicate grapefruit oil was found to repel ants. Thus this reference does not anticipate Claim 1 or new Claim 75.

Moreover, the Mullen patent does not teach or suggest that any of the compounds listed in Claim 1 or nootkatone in Claim 75 would kill or repel ants. It does not discuss any components of any of the oils.

Thus Applicants respectfully submit that the §102 rejections based on the above references should be withdrawn, and that Claims 1-5 and 6-10, as amended, should be allowed. In addition, new Claims 75-77 are not anticipated by any of the references, and thus should also be allowed.

Moreover, none of the above references either individually or together would teach or suggest the invention as claimed in the two independent claims 1 and 75. Grapefruit oil contains 90% limonene which is known to repel insects. See U.S. Patent Nos. 6,130,253 and 5,977,186. A person skilled in the art would neither be motivated nor have any expectation of success to try using nootkatone or  $\alpha$ -cedrene to repel or kill ants. Applicants respectfully submit that none of the above references should be used to reject the claims under either 35 U.S.C. §102(a) or 35 U.S.C. §103(a).

Applicants thus submit that Claims 1 and 75 are neither anticipated nor made obvious over the above references, and are in condition for allowance. The claims that depend from these two independent claims should also be allowed.

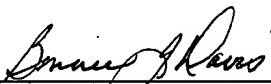
### ***The §103(a) Rejection***

Claim 2 was rejected under 35 U.S.C. §103(a) as being unpatentable under Zobitne *et al.* (as applied to Claims 1, 3-7, and 10) and also Mullen (as applied to Claims 1, 4, 7, and 10 above). Claim 2 relates specifically to fire ants as a subgroup of ants. Claim 2 depends from Claim 1, which as discussed above, should be allowed following the amendment. As discussed above, neither the Zobitne or Mullen patent teach or suggest use of  $\alpha$ -cedrene, zizanol, or bicyclovetivenol as a treatment to repel or kill fire ants.

### **Conclusion**

If any issues arise that may present an obstacle to allowance, the undersigned would welcome a telephone call to discuss such matters before further action is taken. Otherwise, allowance of Claims 1-5, 7-10, and 75-77 at an early date is respectfully requested.

Respectfully submitted,



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# Grapefruit Gland Oil Composition Is Affected by Wax Application, Storage Temperature, and Storage Time

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The effect of wax application, storage temperature (4 or 21 °C), and storage time (14 or 28 days after wax application) on grapefruit gland oil composition was examined by capillary gas chromatography. Wax application decreases nonanal and nootkatone levels.  $\beta$ -Pinene,  $\alpha$ -phellandrene, 3-carene, ocimene, octanol, *trans*-linalool oxide, and *cis*-*p*-mentha-2,8-dien-1-ol levels increase, but limonene levels decrease, with temperature. Levels of  $\alpha$ -pinene, limonene, linalool, citronellal,  $\alpha$ -terpineol, neral, dodecanal, and  $\alpha$ -humulene decrease with time. Levels of  $\alpha$ -phellandrene, 3-carene, ocimene, and *trans*-linalool oxide increase with time. No compound level was affected by the interactive action of temperature and wax application, suggesting that these two factors cause grapefruit oil gland collapse (postharvest pitting) through means other than changing gland oil composition. Compounds that are toxic to the Caribbean fruit fly ( $\alpha$ -pinene, limonene,  $\alpha$ -terpineol, and some aldehydes) decrease with time, thus suggesting grapefruit becomes increasingly susceptible to the fly during storage.

**Keywords:** Gland oil; wax application; temperature; time; postharvest pitting

## INTRODUCTION

Postharvest pitting of white grapefruit is characterized by clusters of collapsed oil glands that develop over the surface of the fruit (Petracek et al., 1995). The collapse is stimulated by wax application and high-temperature storage ( $\geq 10$  °C) common in handling of grapefruit. Disorders with similar morphologies and etiologies have also been observed in Fallglo tangerines (Petracek et al., 1998) and Temple oranges (Petracek et al., 1997). Gland oil constituents are largely terpenes and sesquiterpenes and are highly phytotoxic (Soule and Grierson, 1986). Oil gland collapse may be due to changes inside the gland oil or changes of the surrounding cell walls directly contacting gland oil, either of which may result in increased susceptibility of the surrounding cell walls to gland oil. Because postharvest pitting causes substantial losses during storage of citrus fruit, we have been interested in improving our understanding of peel physiology to help predict or control this disorder.

It has been necessary to subject grapefruit to adverse physical conditions to kill Caribbean fruit fly eggs or larvae present before the grapefruit are exported from Florida to locations such as Japan. Cold storage was recommended as one of such adverse conditions (Ismail et al., 1986). Naturally occurring oxygenated compounds, some of which are present in grapefruit peel oil, have been found to be toxic to Caribbean fruit fly larvae (Davis et al., 1976; Styer and Greany, 1983). Therefore, any change in peel oil might affect the survival of the larvae in grapefruit (Greany et al., 1983).

This paper reports the effects of storage temperature, wax application, and storage time on grapefruit gland

oil composition because of concerns about (1) whether changed gland oil composition is the direct cause of postharvest pitting and (2) whether treatments (waxing, temperature, and time) may affect fruit susceptibility to fruit flies by altering certain compound contents. To serve these purposes, oil taken directly from the oil gland by syringe was analyzed.

## MATERIALS AND METHODS

**Plant Material.** Mature Marsh white grapefruit were harvested from groves in Fort Pierce, FL, on February 8, 1998. Fruit were packed and stored at the Citrus Research and Education Center in Lake Alfred, FL, on February 11. Fruit were washed on roller brushes with FMC Fruit Cleaner 395 (FMC, Lakeland, FL), waxed, and stored at 21 °C. Wax application consisted of coating the fruit with a commercially available shellac-based wax (FMC) on roller brushes and drying the fruit at  $60 \pm 5$  °C for 2 min. Fruit were not degreased with ethylene or treated with fungicide.

**Internal Gas Level.** Grapefruit internal gas level was analyzed as previously described (Petracek et al., 1998). Briefly, septa were created by applying dabs (~1 cm in diameter) of Dow 3140 RTV silicone (Midland, MI) to the stylar end of the fruit. Gas samples (20 mL) were taken from the internal air space of the fruit by syringe. Samples were analyzed by a flow-through system consisting of O<sub>2</sub> (model 26112, Orbisphere Laboratories, Geneva, Switzerland) and CO<sub>2</sub> (model LI-6251, Li-Cor, Lincoln, NE) gas analyzers connected in series with N<sub>2</sub> used as the carrier gas.

**Chromatography.** Samples were analyzed on a Hewlett-Packard 5890 high-resolution capillary gas chromatography with a 0.32 mm i.d.  $\times$  30 m DB-5 column (Restek, Bellefonte, PA). The initial oven temperature was 32 °C, held for 5 min, then ramped at 7 °C min<sup>-1</sup> to 260 °C, and held there for 5 min. The injector temperature was 260 °C, the detector temperature was 260 °C, and the hydrogen carrier gas flow was 50 cm/s. Retention index values were established using standards. Peaks were identified by using retention indices and by mass spectral information obtained from standards and the literature.

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Table 1. Effect of Waxing and Temperature on Internal O<sub>2</sub> and CO<sub>2</sub> Levels of Marsh Grapefruit<sup>a</sup>

	21 °C		4.5 °C	
	no wax	wax	no wax	wax
O <sub>2</sub> (%)	18.0 c	2.5 a	19.2 c	12.1 b
CO <sub>2</sub> (%)	2.1 a	6.2 c	2.1 a	3.2 b

<sup>a</sup> Internal O<sub>2</sub> and CO<sub>2</sub> levels were measured 24 h after waxing (*n* = 10 fruit). Mean separation within columns was by Duncan's new multiple-range tested at  $\alpha$  = 0.05.

**Sample Preparation.** The fluid from three to five intact oil glands was extricated from the middle of the stylar end and equator of the fruit with a syringe (Rouseff et al., 1996) and dissolved in 0.1 mL of methylene chloride. Two microliters of the solution was injected immediately for GC analysis. The punctured part of the fruit was sealed by Dow 3140 RTV silicone. Six fruit were tested for each treatment. The experimental design was three factor with two levels. The parameters are as follows: waxed and not waxed, 4 and 21 °C storage temperature, and 14 and 28 days storage time. Factorial analysis was performed by PlotIT (SPE, Haslett, MI).

## RESULTS AND DISCUSSION

Twenty-seven volatile components in grapefruit oil extracted from the peel were determined as listed in Table 2. Wax application decreased nonanal and nootkatone at 95% level and 99% level, respectively. Nonanal was specifically decreased by waxing after 28 days and at 21 °C. Nootkatone was decreased by waxing at 21 °C but not at 4 °C. Limonene level decreased with temperature. Levels of  $\beta$ -pinene,  $\alpha$ -phellandrene, 3-carene, ocimene, octanol, *trans*-linalool oxide, and *cis*-*p*-mentha-2,8-dien-1-ol increased with temperature. Levels of  $\alpha$ -pinene, limonene, linalool, citronellal,  $\alpha$ -terpi-

neol, neral, dodecanal, and  $\alpha$ -humulene decreased with time. Levels of  $\alpha$ -phellandrene, 3-carene, ocimene, and *trans*-linalool oxide increased with time.

Waxing significantly decreased grapefruit internal O<sub>2</sub> level and increased internal CO<sub>2</sub> level. (Table 1). Shaw et al. (1990) found that a low-oxygen (<1%) controlled atmosphere treatment had little effect on Valencia orange essence oil composition, whereas changes in terpene, alcohol, and aldehyde levels occurred in Pineapple orange essence oil. Because both nonanal and nootkatone are formed through oxidation (Erduran and Hotchkiss, 1995; Wilson and Shaw, 1978; Drawert et al., 1984), lowered internal O<sub>2</sub> by wax application might be responsible for the decrease in their levels.

Linalool was found to be the most phytotoxic compound in citrus oil gland (Wild, 1992). Neither wax application nor high-temperature storage affected its level. Limonene was found to be second in phytotoxicity (Wild, 1992). Wax application did not affect its level, whereas high temperature decreased its level. Neither nonanal or nootkatone, two compounds affected by wax application, was known to possess remarkably high or low phytotoxicity. Moreover, not a single compound level was affected by waxing and temperature interactive effect. Because wax application combined with high-temperature storage stimulates postharvest pitting (Petrcek et al., 1995), it seems that pitting was caused by means other than changing gland oil composition. Changes in oil gland wall composition by wax application and high-temperature storage that impact susceptibility to gland oil may be the cause of postharvest pitting.

Aldehydes are more toxic to Caribbean fruit fly than alcohols and ketones (Davis et al., 1976), which may be

Table 2. Percentage Composition of Marsh Grapefruit Peel Oil Affected by Waxing, Storage Temperature, and Storage Time

compound	index	day 14				day 28				significance <sup>a,b</sup>						
		21.0 °C		4.5 °C		21.0 °C		4.5 °C		w	T	t	wT	wt	Tt	wTt
		no wax	wax	no wax	wax	no wax	wax	no wax	wax							
$\alpha$ -pinene	938	0.62	0.627	0.63	0.62	0.60	0.614	0.61	0.612			*				
sabinene	976	0.77	0.723	0.688	0.693	0.850	0.777	0.540	0.677							
$\beta$ -pinene	980	0.08	0.079	0.067	0.063	0.093	0.084	0.061	0.067		*					
myrcene	991	1.94	1.912	1.939	1.948	1.932	1.931	1.893	1.911							
octanol	1004	0.67	0.765	0.649	0.734	0.710	0.670	0.660	0.509							
$\alpha$ -phellandrene	1009	0.07	0.057	0.076	0.075	0.190	0.173	0.134	0.096							
3-carene	1023	0.04	0.050	0.053	0.050	0.256	0.272	0.194	0.151							
limonene	1039	92.63	92.25	92.943	92.949	89.083	90.853	92.668	92.467							
ocimene	1051	0.27	0.305	0.223	0.187	0.384	0.238	0.273	0.281							
$\gamma$ -terpinene	1067	0.06	0.064	0.056	0.037	0.170	0.133	0.108	0.103							
octanol	1074	0.03	0.048	0.019	0.020	0.042	0.020	0.010	0.020		*					
<i>cis</i> -linalool oxide	1082	0.03	0.043	0.030	0.036	0.028	0.018	0.010	0.054							
<i>trans</i> -linalool oxide	1099	0.07	0.071	0.075	0.070	0.328	0.307	0.213	0.180							
linalool	1107	0.10	0.084	0.076	0.083	0.050	0.054	0.042	0.089							
nonanal	1110	0.11	0.096	0.099	0.109	0.148	0.101	0.125	0.092							
<i>cis</i> - <i>p</i> -mentha-2,8-dien-1-ol	1149	0.07	0.078	0.089	0.095	0.239	0.174	0.051	0.023		*					
citronellal	1161	0.05	0.049	0.063	0.056	0.038	0.034	0.040	0.054							
$\alpha$ -terpineol	1210	0.05	0.046	0.031	0.036	0.011	0.018	0.009	0.023							
decanal	1216	0.50	0.514	0.480	0.501	0.495	0.514	0.499	0.469							
neral	1258	0.05	0.051	0.046	0.043	0.032	0.032	0.041	0.032							
$\alpha$ -copaene	1418	0.084	0.095	0.084	0.0908	0.0901	0.0900	0.09	0.0900							
dodecanal	1419	0.11	0.097	0.098	0.092	0.069	0.071	0.078	0.084							
<i>trans</i> -caryophyllene	1456	0.17	0.172	0.168	0.171	0.178	0.165	0.158	0.172							
$\alpha$ -humulene	1491	0.03	0.033	0.030	0.027	0.023	0.020	0.024	0.026							
$\gamma$ -cadinene	1502	0.11	0.106	0.112	0.111	0.111	0.114	0.117	0.123							
nootkatone	1847	0.43	0.282	0.297	0.282	0.636	0.309	0.315	0.322		**	*				
total		99.12	99.025	99.204	99.264	96.883	97.87787	99.052	98.816							

<sup>a</sup> Factors w, T, t, wT, wt, and Tt represents waxing, temperature, time, waxing interacting with temperature, waxing interacting with time, and temperature interacting with temperature, respectively. <sup>b</sup> \*, \*\*, and \*\*\* represent confidence levels of 95%, 99%, and 99.9%, respectively.

due to the role of aldehydes as potent uncouplers of oxidative phosphorylation (De Greef and Van Sumere, 1966). Three of five aldehydes listed in Table 2 decreased with time. In addition,  $\alpha$ -pinene, limonene, and  $\alpha$ -terpineol are the three compounds that showed toxicity to Caribbean fruit fly in Styer and Greany's (1983) work, and all of the three compounds decrease with time (Table 2). Therefore, grapefruit might become increasingly susceptible to Caribbean fruit fly with time in storage.

Nootkatone levels increased with storage time but were reduced by wax application and cold storage (Table 2). Wilson et al. (1990) found that increases in nootkatone content in grapefruit oil with maturity paralleled those reported in navel orange for valencene, the level of which changed concomitantly with biochemical and physiological changes associated with senescence (Coggins et al., 1969). Nootkatone is probably formed in vivo from valencene through 2-hydroxyvalencene (nootkatol), because several cell suspension cultures from citrus were able to convert valencene to nootkatone (Drawert et al., 1984). Because the level of valencene in grapefruit is low and thus difficult to quantify, nootkatone may serve as a senescence indicator or predictor in grapefruit. It is interesting that for all compounds levels affected by both temperature and time, including  $\alpha$ -phellandrene, 3-carene, limonene, ocimene, *trans*-linalol oxide, and nootkatone, higher temperature and longer time have the same effect (increase or decrease). Each of these compounds may be a candidate for senescence indicator or predictor because longer storage time and/or higher temperature promote senescence. On the other hand, because wax application and cold storage can decrease nootkatone level, both might be used to slow grapefruit senescence.

Waxed and stored grapefruit may be processed into juice. The peel oil in grapefruit contributes to the characteristic flavor of grapefruit juice because some oil is introduced into the juice during extraction. Among compounds that show treatment effects (Table 2), nootkatone and limonene are most important to grapefruit juice flavor, whereas  $\alpha$ -terpineol contributes negatively to grapefruit flavor (Pino et al., 1986). Nootkatone has a typical grapefruit aroma and exhibits a low odor threshold (Berry et al., 1967). It is believed to be a major flavor impact compound (MacLeod and Buigues, 1964).

#### ACKNOWLEDGMENT

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